The Flotation Behavior of Digested Asphalt Ridge Tar Sands

R. J. Smith and J. D. Miller

Abstract—*The hot water process for Utah tar sands differs significantly from that used for Canadian tar sands due to inherent differences in respective bitumen viscosities and the nature of bitumen-sand association. These differences have led to the identification of certain design criteria necessary to achieve satisfactory phase disengagement during digestion (Sepulveda and Miller, 1978; Misra and Miller, 1980). Subsequent flotation of digested tar sand is a subject of current attention and is considered in this publication for the Asphalt Ridge deposit.*

Although contact angle measurements of solvent extracted Asphalt Ridge bitumen indicated moderate hydrophobicity, air bubble attachment to the bitumen concentrate is not possible. This surprising result suggests that flotation separation is dependent on air bubble entrapment rather than on air bubble attachment due to surface hydrophobicity. The entrapment of air bubbles in the bitumen was apparent from examination of the bitumen concentrate, especially at lower flotation temperatures.

A factorial design of the major operating variables in flotation separation indicated that the quality of separation was significantly dependent on the flotation temperature and to a lesser extent on the degree of agitation. For a flotation temperature of 77°C a recovery of 96.7% was realized at a grade of 61.0% bitumen. The improved separation at higher flotation temperatures was found to be due to the decrease in bitumen viscosity resulting in more effective rejection of coarse sand from the concentrate.

This research effort, which has been sustained for the past six years at the University of Utah, is being continued to provide necessary design data for the construction of a pilot plant to treat Utah tar sands.

Introduction

The purpose of this paper is to report results of research conducted at the University of Utah on the separation of bitumen from Utah tar sands by a hot water processing technique to produce a feedstock for synthetic fuel and petrochemical production.

It is understood that the term tar sands refers to a naturally occurring sandstone impregnated with a highly viscous crude hydrocarbon, a bitumen (Perrine, 1975). Early observers named this material tar sand after the asphaltic appearance of the deposit. Geologists referred to these same deposits as oil sands because they represented a potential source of oil. Generally, the sand component of tar sands is mostly quartz with some minor clay and calcareous constituents.

Major tar sand deposits occur in Canada, Venezuela, and the US. The largest of these are the Canadian deposits which contain 143 km^3 of crude, in-place bitumen. This is the only deposit in the world being mined and processed commercially to recover bitumen for the production of synthetic crude oil. It is important at this point to distinguish between the processing strategy used to separate bitumen from the Athabasca tar sands and the University of Utah hot water process for Utah tar sands.

Processing Athabasca Tar Sands

Dr. K. A. Clark working for the Alberta Research Council is credited with first describing the hot water process for Athabasca tar sands in 1923. The process he proposed has since undergone much evaluation and modification, but contains the essential elements on which today's commercial ventures are based. In 1944, Dr. Clark set forth the fundamental features that characterize the hot water process (Clark, 1944). From these fundamental features, several different process strategies were investigated, culminating in the development of plants by GCOS (Great Canadian Oil Sands Ltd.) and Syncrude Canada, Ltd. Both plants have similar flow-sheets for bitumen recovery as described by Camp (1974) and shown in Fig. 1. Mining is accomplished with large bucket wheel excavators and/or drag lines. The tar sand is then conveyed by a belt to the plant where it is fed to a rotating drum and mixed with hot water and steam. The tar sand lumps are reduced in size by ablation as each lump is warmed and surfaces slough off. Sodium hydroxide is added as a process aid to control pH. The residence time in the rotary drum is sufficient to accomplish the required heat transfer to disintegrate each lump by heating and mechanical mixing. As a result bitumen is displaced from sand particles and pulp is discharged at about 85°C. The conditioned tar sand is then screened to remove tramp materials and tar sand lumps.

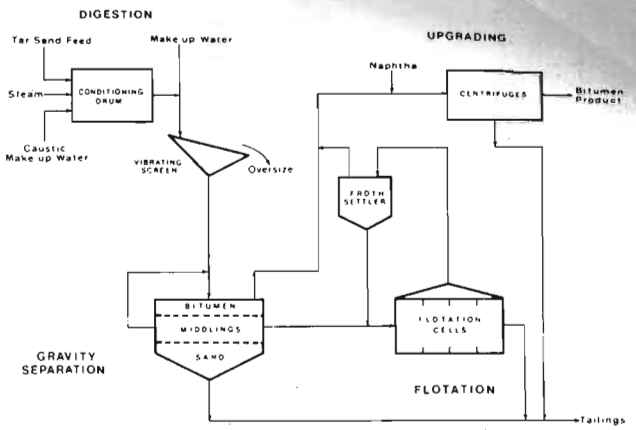

Fig. 1—Hot water process for Athabasca tar sand.

Phase separation of bitumen from sand is initially accomplished in a gravity settler, which is essentially a modified thickener. Tar sand is introduced into the feed well and bitumen floats to the surface of the settler where it is removed by radial arms. Most sand sinks to the bottom conical section of the thickener, is advanced to the center by rakes, and then discharged as a tailing product. From the side of the thickener a middling stream is taken, which contains some fine mineral and bitumen particles. A portion of this stream is returned to the rotating drum for pulp density control (Hendrickson, 1975). The balance of the middling stream is fed to standard flotation cells, that can be either tank type or trough type (Porteous et al., 1979).

Bitumen concentrate from the gravity settler and the scavenger flotation cells are combined and further treated and

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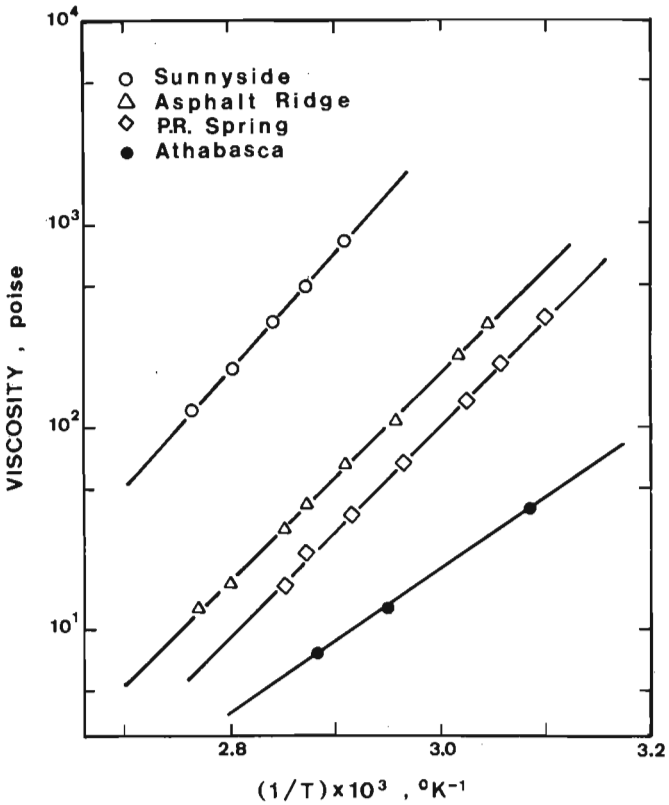

Fig. 2—Arrhenius-type plot illustrating the effect of temperature on viscosity for bitumen from various tar sand deposits.

upgraded to a synthetic crude oil. Bitumen concentrate produced in this manner contains about 84% bitumen and 16% mineral matter on a dry basis, with a bitumen recovery of 90%. Hot water processing, as described above, proved to be quite successful in processing Athabasca tar sands. Unfortunately this process is not so successful when applied to Utah tar sands.

Processing Utah Tar Sands

Utah Geological and Mineral Survey published a paper by H. R. Ritzma (1973) in which he states there are 51 tar sand deposits in Utah. The 25 deposits in the Uinta Basin contain an estimated 1.7 km^3 of in-place bitumen. Utah's reserves contain 4-4.7 km^3 of bitumen representing about 96% of the bitumen saturated sandstone in the US.

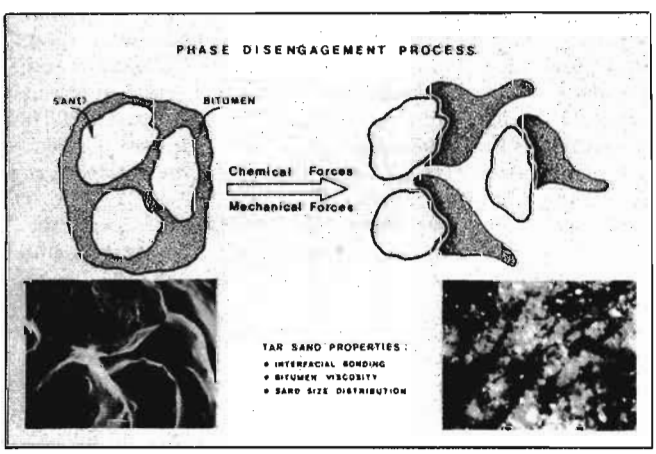

Fig. 3—Phase disengagement process including a photomicrograph of digested asphalt ridge tar sand.

A deposit containing 159 hm^3 (one billion barrels) is sufficient to produce 16 dam^3/d of synthetic crude oil for 20 years, based on a 70% yield of synthetic crude oil from bitumen (Oblad, 1977). This yield is not unreasonable as indicated from the GCOS operating experience. Unfortunately, the process used for Athabasca tar sands does not result in an effective bitumen-sand separation when directly applied to Utah tar sands.

The characteristic differences between Athabasca and Utah tar sands have been chronicled in two previous papers from the Metallurgy Department at the University of Utah (Sepulveda and Miller, 1978; Misra and Miller, 1980). Unlike Athabasca tar sand, Utah tar sands contain no detectable indigenous water. Therefore, bitumen is bonded directly to sand particles. Another difference between Athabasca and Utah tar sands is the viscosities of Utah bitumens appear significantly greater than viscosities of Athabasca bitumen. For example, reported viscosity of Asphalt Ridge bitumen are compared with viscosity for Athabasca bitumen in Fig. 2.

Armed with this information concerning the differences between Athabasca and Utah tar sands, a hot water process suitable for Utah tar sands was developed. The cirteria for effective phase disengagement during digestion were found to be a high shear force field, an appropriate addition of alkali wetting agent, and temperatures near the boiling point of water. In the case of tar sands containing highly viscous bitumen, such as the Sunnyside deposit, the addition of diluent may be necessary. The phase disengagement process is presented in Fig. 3, which includes a photomicrograph of digested Sunnyside tar sand. It was found that effective phase separation could not be achieved with a gravity settler because sand entrained by bitumen resulted in an agglomerate having a higher density than water, hence the bitumen would not float. As a result, a flotation technique was used to effect phase separation and assess the quality of phase disengagement. These previous studies focused on determining the optimum operating conditions for digesting Utah tar sands in an electrically heated stirred tank reactor. It is the purpose of this paper to report new experimental results regarding the flotation behavior of digested Asphalt Ridge tar sand.

Experimental Methods

Two types of experiments were used to evaluate and describe the flotation behavior of digested Asphalt Ridge tar sand. Literature extensively discusses the natural hydrophobicity of bitumen as well as air bubble/bitumen droplet collision and attachment phenomena (Bowman, 1970). In this regard, contact angle measurements were made at the bitumen surface as a function of temperature and aqueous phase composition. Also, experiments were done to study operating variables in the flotation step of the hot water process and determine their effect on the efficiency of phase separation.

Contact Angle Measurements

Contact angle measurements were made using a goniometer and optical bench manufactured by Rame-Hart. A special feature of the optical bench is an environmental chamber that allows for system temperature control. A microsyringe was used to dispense air bubbles.

To measure a contact angle, the environmental chamber was filled with distilled water and the contoller was set for the desired temperature. The bitumen sample was placed in a plastic bottle cap and allowed to form a smooth surface. The cap was then weighted with a steel ring and placed in a 20 mm × 20 mm × 40 mm cuvette to which the desired solution was then added. The cuvette was then placed in the environmental chamber and allowed to equilibrate at the desired temperature. Then air bubbles were touched to the bitumen surface. When attachment occurred, the contact angle was measured.

Hot Water Processing Experiments

Two major steps in the hot water process are phase disengagement followed by flotation. The digester is a 3.8 L stirred-tank reactor with two opposing pitch blade impellers, an SCR speed controller, a heating mantle with temperature controller, and a tachometer. Flotation is conducted using a 38 L cell, made by Hazen Research Inc., similar in design to Denver Laboratory units. The cell is equipped with a urethane impeller and stator. Air is injected through the impeller shaft at a flowrate measured with a Gilmont rotameter. Impeller speed is set with an adjustable motor pulley sheave. In addition, the cell is equipped with a 38 mm bottom discharge ball valve. An advantage of this cell is that the impeller and motor can be raised and the cell tilted in its frame for easy cleaning.

To conduct a batch experiment, 1.5 L of 0.3 M solution of sodium carbonate is put into the reactor and raised to temperature with the heating mantle. After the solution is heated to about 80°C, 4 kg of tar sand, reduced in size to minus 9.5 mm, is fed into the reactor through a feed port. During this time, impellers rotate at 200-250 rpm to prevent plugging of the discharge port. When feeding is completed, pulp temperature in the reactor usually has reached 95°C, the desired digestion temperature. The tar sands are then digested at 750 rpm for 15 minutes.

After digestion, pulp is discharged into the flotation cell operating at a selected rpm and air flowrate. The cell contains about 34 L of water at the desired temperature and a specified amount of sodium carbonate. Bitumen floats immediately and is removed for three minutes, which is sufficient time to remove all but very slow floating bitumen. Bitumen-free sand is not hydrophobic and does not report to the concentrate. Tailings are discharged out the bottom valve of the cell at the end of each batch test.

Samples of feed, concentrate, and tailings are analyzed to determine bitumen, water, and sand content with a Dean Stark apparatus, according to the procedures reported by the US Bureau of Mines (Rall and Raliaferro, 1946). This toluene extraction method was well described in earlier reports.

Results and Discussion

Contact Angle Measurements

Bubble-particle attachment is considered one key factor controlling the froth flotation process. The thermodynamic considerations of bubble-particle attachment have been discussed at length by several authors (Finch and Smith, 1979; Laskowski, 1974; Fuerstenau and Raghavan, 1976). Even without consideration of heats of immersion, heats of adhesion, heat of bubble deformation and interfacial tensions, which are involved in a detailed analysis of the attachment process, the contact angle is still a good indication of the hydrophobicity or degree of surface wetting.

For Asphalt Ridge feed samples equilibrated with sodium carbonate solutions of pH 9 and pH 11 at 25°C, contact angles of 34° and 39° were observed. These results indicate a moderate hydrophobic character of the bitumen attached to sand particles, and may reflect the heterogeneous nature of the surface.

Two samples of benzene-extracted bitumen were then tested. The first sample was Asphalt Ridge bitumen from feed material used by Sepulveda and Miller (1978). Contact angles in sodium carbonate solutions of pH 9 and pH 11 were found to be 76° and 75°. The second sample was bitumen extracted from the Asphalt Ridge feed material being used for the present study. In sodium carbonate solutions of pH 9 and pH 11, the measured contact angles were 60° and 69°, respectively. In addition to contact angle measurements at 25°C, measurements were made at 35°C and 45°C, and the initial contact angles were essentially the same as those measured at 25°C.

Next, to examine the hydrophobic nature of the bitumen concentrate, a sample from a hot water processing experiment was taken and a contact angle measurement was immediately attempted. At both pH 9 and pH 11 a bubble would not attach to the surface of the bitumen concentrate. This result is somewhat surprising because it had been assumed bubble attachment to bitumen droplets was an important aspect of the flotation step. Some yet unpublished work by Miller and Misra (1981) shows that naturally occurring, untreated Sunnyside bitumen, found oozing out of the deposit, exhibits a natural hydrophobic character at a pH of 7.2, similar to the benzene-extracted bitumen; however, at higher pH values air bubbles would not attach to the bitumen surface.

Some tar sand researchers have observed air bubble-bitumen droplet attachment in a flotation cell, while others postulate that a bitumen film rapidly envelops the air bubble thus accounting for bitumen flotation (Bowman, 1970). Such phenomena involved in the flotation process are revealed in Fig. 4, which shows a time sequence of photographs of an air bubble (1.0 mm diam) attached to the surface of a toluene-extracted bitumen at 45°C in a pH 11.0 sodium carbonate solution. Note how bitumen seems to wet the air bubble and lower viscosity at this temperature permits the buoyant force to stretch the bitumen resulting in failure after several minutes. This observation may help to explain the flotation response variation with temperature, as discussed later.

Hot Water Processing Experiments

Following the work of Sepulveda and Miller (1978), which identified those conditions most favorable for the digestion of Asphalt Ridge tar sand, the objective of this phase of the research project was to identify those flotation variables and conditions resulting in the most favorable phase separation. Traditionally, it is recognized that the degree of agitation and air flowrate are always important variables in a flotation process. From preliminary flotation tests it was noted that the temperature and sodium carbonate concentration also appeared to have an effect on the efficiency of bitumen recovery by flotation.

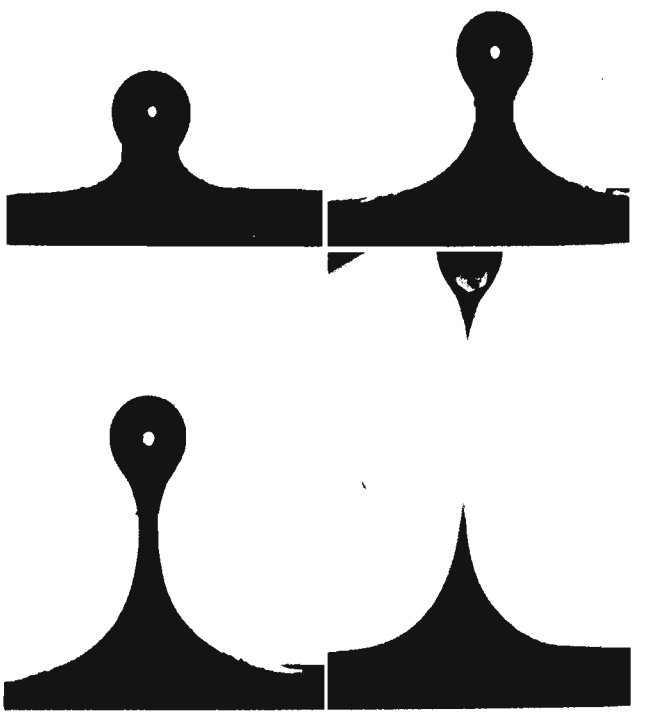

Fig. 4—The dynamic behavior of an air bubble attached to an asphalt ridge, benzene-extracted, bitumen surface at 45°C.

Table 1—Variable Ranges for First Order Factorial Design (2^4)

Variable		Units	Coded Values −1	0	+1
Temperature	(X_1)	°C	30	50	70
Air Flow Rate	(X_2)	L/min	5	15	25
Agitation	(X_3)	rpm	600	800	1000
Na_2CO_3	(X_4)	mol/L	0.025	0.05	0.075

To test the effect of these four variables, a two level, four variable factorial design with center point replication was used. The coefficient of separation was selected as the objective function to be maximized. The coefficient of separation (CS) is a one-parameter description of the efficiency of separation for a two-component system, and physically represents the fraction of feed material that undergoes perfect separation. Thus, an ideal separation would be represented by a coefficient of separation of unity, whereas if no separation is achieved the coefficient of separation would be zero. The coefficient of separation takes into consideration both grade and recovery and can be shown to be equal to the recovery of the valuable constituent in the concentrate minus the recovery of the gangue in the concentrate.

Table 1 presents the variables and their ranges used in this series of experiments, while the experimental results are tabulated in terms of the coded variable level and the coefficient of separation in Table 2. These values were used for the determination of a first order model of the flotation response surface of the following form:

$$y = B_o + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 \qquad (1)$$

Multiple linear regression of this data allowed for the evaluation of the coefficients and equation (1) becomes

$$CS = 0.8378 + 2.5\times10^{-2}X_1 - 1.13\times10^{-4}X_2 + 1.00\times10^{-2}X_3 - 2.75\times10^{-4}X_4 \qquad (2)$$

From hypothesis testing, it was found that this model describes the data within experimental error and represents a planar response surface. Analysis of variance and further hypothesis testing indicates that the effect of air flowrate and sodium carbonate are not statistically significant so B_2 and B_4 can be taken as equal to zero. The reduced model

$$CS = 0.8378 + 2.50\times10^{-2}X_1 + 1.00\times10^{-2}X_3 \qquad (3)$$

also describes the data within experimental error. The effect of these process variables were further considered with respect to results obtained from the factorial design.

Table 2—Experimental Conditions and Coefficients of Separation for the 2^4 Factorial Design

Experiment No.	X_1	X_2	X_3	X_4	Coefficient of Separation
1	−1	−1	−1	−1	0.6224
2	1	1	−1	−1	0.8495
3	1	−1	1	−1	0.8773
4	−1	1	1	−1	0.8214
5	1	−1	−1	1	0.6757
6	−1	1	−1	1	0.7827
7	−1	−1	1	1	0.8206
8	1	1	1	1	0.6795
9	1	−1	−1	−1	0.8415
10	−1	1	−1	−1	0.8012
11	−1	−1	1	−1	0.8270
12	1	1	1	−1	0.8745
13	−1	−1	−1	1	0.7968
14	1	1	−1	1	0.8625
15	1	−1	1	1	0.8522
16	−1	1	1	1	0.8404
17	0	0	0	0	0.8254
18	0	0	0	0	0.8292

Table 3—Influence of Flotation Temperature on the Coefficient of Separation

Operating Conditions:		Air Flowrate Agitation Na_2CO_3	15 L/min 1000 rpm 0.05M	
Temperature (°C)	Conc. Grade (% Bitumen)	Bitumen Recovery (%)	Coefficient of Separation Experimental	Predicted
0	46.0	91.4	0.78	0.79
1	47.3	95.4	0.81	0.79
35	48.7	97.0	0.82	0.83
50	57.3	96.9	0.87	0.85
57	61.9	96.6	0.88	0.86
77	61.0	96.7	0.89	0.88

Temperature Effect

As described, temperature appeared to be the most significant variable. In this regard it was decided to measure the efficiency of separation as a function of flotation temperature only. To do this the three other independent variables were set at constant conditions; air flowrate of 15 L/min ($X_2 = 0$), agitation of 1000 rpm ($X_3 = 1$), and sodium carbonate concentration of 0.05M ($X_4 = 0$). The model equation then becomes:

$$CS = 0.8478 + 2.5\times10^{-2}X_1 \qquad (4)$$

The flotation response was studied for flotation temperatures 0°C to 77°C. Experimental results are summarized in Table 3 and graphically displayed in Fig. 5. This data shows only fair agreement between experimental and predicted results. It can be seen, as expected, that as temperature of flotation increases the coefficient of separation improves significantly. From Table 3 it can be noted that the increase in coefficient of separation is a result of both an increase in concentrate grade and increased bitumen recovery. The data also suggest a nonlinear increase in the coefficient of separation between 40°C and 60°C that the first order model would not be able to predict. This nonlinearity may reflect the change in bitumen viscosity or the change in interfacial tensions in the four phase system (bitumen, water, sand, and air).

During this testing phase the different nature of bitumen concentrates was observed as a function of temperature. Below about 50° to 55°C the concentrate is spongy in structure, an agglommeration of bitumen and air. This structure is stable and will float in water even after removal from the flotation cell.

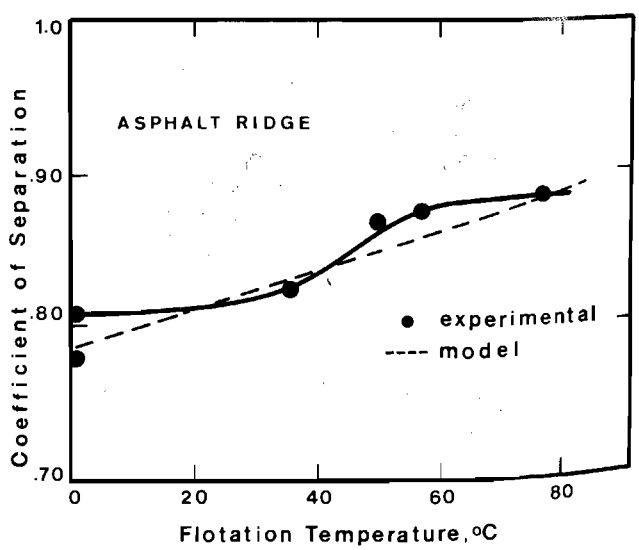

Fig. 5—Influence of flotation temperature on the coefficient of separation at 0.05M Na_2CO_3 and 1000 rpm.

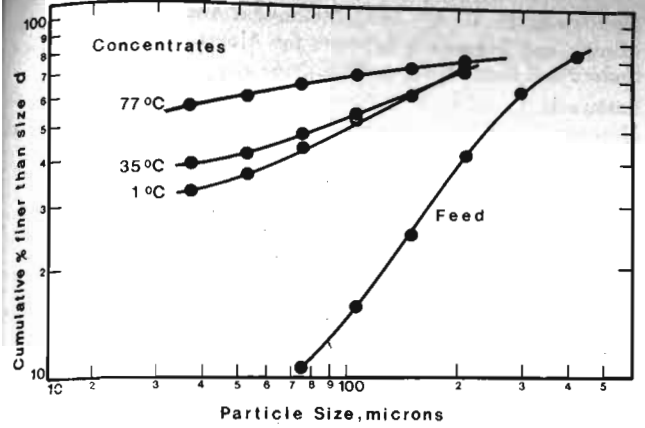

Fig. 6—The effect of flotation temperature on the size distribution of sand entrapped in the bitumen concentrate.

Above 55°C the bitumen concentrate floats on the surface of the pulp due to buoyant forces of bubbles, which readily pass through the low viscosity bitumen and give the bitumen an appearance of boiling. After removal from the air source, all air bubbles rapidly escape and the biumen's spongy structure collapses. In effect, above 50° to 55°C, the bitumen behaves more like a liquid phase than like a solid phase. These observations seem to be supported by the results presented in Fig. 3, which gives evidence for the movement of air bubbles in the bitumen phase.

Concentrate Particle Size

Results indicate that at higher flotation temperatures the less viscous bitumen may allow for better rejection of sand from the bitumen-air aggregate, and conversely at lower flotation temperatures entrapment of coarse sand in the bitumen may account for the poorer concentrate grade. To further examine this effect, the concentrates obtained at different temperatures were extracted to recover sand for size analysis. Size distribution of concentrates was found to become finer with increasing flotation temperature. In other words, higher temperature flotation results in improved rejection of coarse sand. Sand size distributions for concentrates obtained at various flotation temperatures are shown in Fig. 6.

These results confirm the hypothesis regarding improved sand rejection at higher temperatures. Intuitively, such sand rejection is expected due to inertial considerations. Less viscous bitumen does not have the strength to entrap larger particles just as it does not entrap and hold air bubbles, as discussed earlier.

Effect of Carbonate Concentration

The next aspect of the flotation study was further examination of the effect of sodium carbonate addition. Recall that it was shown that carbonate dosage level was statistically insignificant to the quality of separation with the variable range tested (0.025M to 0.075M sodium carbonate). Consideration of the hot water process reveals the possible difficulty and undesirability of operating the digestion step at 0.3M sodium carbonate and the flotation step at 0.025M to 0.075M sodium carbonate. To do so may complicate carbonate concentration control in each step and the water recycling scheme in a continuous process. A disadvantage to constant carbonate concentration is that such an operating condition might result in higher reagent consumption.

Tests were conducted with carbonate levels from 0 to 0.3M, based on the argument that from a process design standpoint flotation at higher carbonate additions may have certain advantages. Results of these tests are listed in Table 4. Note both changes in concentrate grade and bitumen recovery. It is seen that as carbonate dosage increases the coefficient of separation decreases. Some interesting things were observed in going from no carbonate addition to 0.3M sodium carbonate.

With no sodium carbonate (distilled water at almost neutral pH) the bitumen concentrate was very sticky and difficult to handle in that it tenaciously attached itself to everything. The undesirability of such a condition is obvious, but difficult to quantify and has been observed previously (Miller and Sepulveda, 1978). At 0.05M the best recovery and coefficient of separation were obtained. At 0.15M and 0.30M the quality of separation decreased. More importantly two phenomena were observed at these higher carbonate levels. First, a voluminous froth occurred after most of the bitumen was removed from the cell. This froth carried both a bitumen film and a large amount of fine sand. Second, at higher carbonate levels, slow floating bitumen droplets that were not recovered in the three minute flotation time were found remaining in the pulp. These droplets did not appear to coalesce as readily as the bitumen droplets did at lower carbonate concentrations. This behavior can be related to observations made by Misra and Miller (1980), who noted at high additions of sodium carbonate during digestion that the system tended to emulsify. The same, yet less extreme, behavior may occur in the flotation step at high sodium carbonate concentrations.

These results point to a need to learn how to monitor sodium carbonate concentration in this process. It also establishes the need to operate flotation at a lower sodium carbonate concentration (0.05M), while maintaining digestion at a much higher carbonate level (0.3M). The disparity in concentration must also receive some attention in the design of a continuous system.

Summary

These results on the flotation behavior of digested Asphalt Ridge tar sand reveal some important features not previously recognized. From efforts to determine the hydrophobic nature of bitumen surfaces by contact angle measurements, it was found that bitumen exhibited moderate hydrophobicity that, for certain samples, could be destroyed in alkaline solutions. Further, examination of the bitumen concentrate from a hot water processing experiment indicated that it was not hydrophobic even though it had been recovered in the froth phase. This evidence along with observations concerning the structural nature of the bitumen concentrate led to the hypothesis that the bitumen-air aggregate forms in the flotation cell due to air bubble entrapment rather than due to the air bubble being attached at the bitumen surface.

From the factorial design study of the following flotation variables—temperature, rpm, air flowrate, and carbonate addition—it was found, for the range of the variables studied, that a first order model described the data within experimental error. Also, it was found from analysis of variance and hypothesis testing that the quality of separation was independent of air flowrate and carbonate addition under these conditions.

Additionally, it was found that the flotation temperature was the most significant operating variable and that increased tem-

Table 4—Effect of Sodium Carbonate Concentration on the Coefficient of Separation

Operating Conditions:	Temperature	60°C
	Air Flowrate	15 L/min.
	Agitation	1000 rpm

Sodium Carbonate (M)	Conc. Grade (% Bitumen)	Bitumen Recovery (%)	Coefficient of Separation
0	60.5	95.6	0.87
0.05	61.9	96.6	0.88
0.15	62.1	94.5	0.87
0.30	58.2	94.0	0.85

perature improves the recovery and grade of the bitumen concentrate. At higher flotation temperatures the concentrate loses its spongy structure and becomes more like a second liquid phase. Further, the sand from high temperature concentrates was found to have a finer size distribution due to the enhanced rejection of coarse particles from the less viscous bitumen.

The addition of more than about 0.075M sodium carbonate results in voluminous unmanageable froths. Higher additions also tend to stabilize slow floating bitumen droplets that resist coalescence with other droplets. Generally, higher carbonate additions to flotation were found to decrease separation quality. This information on carbonate balance is particularly important for the design and operation of a continuous flow hot water process.

Acknowledgements

The authors wish to acknowledge the invaluable assistance of M. Misra, H. Smith, and J. E. Sepulveda. Financial support of this research project by the HEW Domestic Mining and Mineral Fuels Conservation Fellowship and the Department of Energy Grant No. EY 77-S-03-1762 has contributed significantly to its success.

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Conversions

Convert From	To	Divide By
m^3	bbl	0.159
liter (L)	gal	3.785
mm	in	25.4
L/min	gal/min	3.785